Patterns of Textural Changes in Brittle Cellular Cereal Foods Caused by Moisture Sorption[1]

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ABSTRACT

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The irregular compressive force-deformation curves of two puffed snacks and two types of croutons at various water activities (a_w) were fitted with a polynomial equation. The values of the fitted force at 20 and 30% deformation were measures of these materials' stiffness. The degree of jaggedness of the normalized mechanical signatures, created as a plot of the residuals divided by the corresponding fitted force values, was quantified in terms of an apparent fractal dimension and the mean magnitude of the Fourier power spectrum parameters whose value appears to be associated with brittleness and crunchiness. Plots of the magnitude of the two jaggedness parameters versus a_w had a typical sigmoid shape that could be described by the original or shifted Fermi equation with similar characteristic constants. The relationship between the stiffness parameters and a_w either had the typical Fermian sigmoid shape or had a peak at an intermediate a_w. This suggests that partial plasticization that reduced brittleness could also reduce fragility. In all four products, the characteristic a_w level for jaggedness loss differed from that of the stiffness loss. This observation, which is in agreement with previous reports, indicates that different textural attributes need not change in unison as a result of moisture sorption, and therefore, that a transition from a glassy to a rubbery state can have a different manifestation in different mechanical properties.

The familiar effects of moisture on the texture of cereals and starchy snacks have been well documented (Katz and Labuza 1981; Sauvageot and Blond 1991; Barrett et al 1992, 1994). They are primarily manifested in the character of the force-deformation relationship and the nature of the acoustic signature (which will not be discussed here). Upon moisture sorption, a material that is hard and brittle (or crunchy and crispy) when dry becomes soft and ductile and loses its crunchy and crispy characteristics. The process has been attributed to a glass transition of the material triggered by lowering the glass transition temperature, T_g, to below the ambient temperature (Slade and Levine 1993). Support for this mechanism comes from the known plasticizing effect of water on a variety of bio and synthetic polymers, which is indeed manifested by lowering the T_g.

The glass transition in many synthetic and biopolymeric materials can occur gradually over a fairly broad temperature range on the order of tens of degrees C. Therefore, it is difficult to identify a single temperature below and above which the properties of the material are qualitatively different, as has frequently been claimed in the polymer and food literature. Moreover, identification of the transition in both bio and synthetic polymers by different methods, or even by the same method under different experimental conditions (e.g., DMA at different heating and cooling rate or frequency) can result in discrepancies of up to 10–40°C (Jankowsky et al 1994, Rodriqez 1994). This has led the organizers of a recent ASTM symposium on the issue to conclude that (R. P. Tye in Seyler 1994) "glass transition is a phenomenon that occurs over a temperature range. The range can cover an interval of varying limits from a few to many tens of degrees Celsius, highly dependent on the behavior and history of a particular material or material type. Furthermore, the definition of the range is affected by numerous experimental parameters dependent upon the particular technique used to study the phenomenon. It is clear that there is no single measured glass transition temperature for a material and that the onset and end of the regime cannot be clearly defined in terms of a sharp transition. A better practice would be to use the term assigned glass transition temperature."

Similar concerns have also been raised regarding the glass transition in biopolymers and food materials (Peleg 1994a,b, 1995). They have led to the development of a model, based on Fermi's function, that describes the changes in mechanical properties at and around the transition in terms of two parameters, one to specify its center (temperature, moisture contents or water activity [a_w]), and the other the steepness of the loss of stiffness, strength, brittleness, etc. The model was applied to the loss of stiffness and the smoothing of the force-deformation relationships of two starchy snacks exposed to moist environment, and it was shown that it could be affected differently (Wollny and Peleg 1994). Similar observations were also reported by Attenburrow and Davies (1993), and they too suggest that like in synthetic polymers, the T_g of cereal foods is not a unique temperature but one that depends on the property used for its determination. More recently, Nicholls et al (1995) also showed that the glass transition has a different effect on different mechanical properties and that therefore the brittleness of biopolymers cannot be predicted on the basis of known T_g alone. The evidence that mechanical properties of cereals exposed to moisture need not change in unison has been rather sketchy. The objectives of this work are to analyze the mode by which a_w affects two mechanical characteristics: the stiffness and the jaggedness of the force-deformation relationship, in terms of a mathematical model previously used by Wollny and Peleg (1994) and to modify the model so that it could account for relationships not found in their work.

MATHEMATICAL MODEL

The relationship between mechanical, or textural, parameters and a_w can assume various shapes as shown in Figure 1. The most frequently encountered type has a characteristic sigmoid shape that can be described by the Fermi function (Peleg 1994a–c, Wollny and Peleg 1994) which for our purpose becomes:

$$Y(a_w) = Y_o/\{1 + \exp[(a_w - a_{wc})/b]\} \quad (1)$$

where $Y(a_w)$ is the magnitude of the mechanical parameter (stiffness, crunchiness); Y_o is its magnitude in the dry state; a_{wc} is a characteristic a_w where $Y(a_{wc}) = Y_o/2$; and b is a constant that

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accounts for the steepness of the relationships around a_{wc}. According to this model, when $b \rightarrow 0$, $Y(a_w)$ approaches a step function, while a large value presents a broad transition. There are parameters, however, that theoretically cannot be smaller than a certain value. A notable example is the apparent fractal dimension of a line (D_f,) whose theoretical range is $1 < D_f < 2$ by definition (see below). In such a case, or where the plasticized wet material still retains a considerable degree of stiffness or strength, the model can be modified to account for the residual magnitude (Wollny and Peleg 1994):

$$Y(a_w) = (Y_o - Y_r)/\{1 + \exp[(a_w - a_{wc})/b]\} + Y_r \quad (2)$$

where Y_r is the residual stiffness, strength, etc. Both Eqs. 1 and 2 entail that at low a_w levels, before plasticization, $Y(a_w)$ is either parallel to the a_w axis or has a negative slope. It is possible, however, that the relationship between certain parameters and a_w has a region in which it has a positive slope (Fig. 1, bottom). If the slope is small it can be ignored. Otherwise, it requires a modification of the model. The simplest is through an added linear term:

$$Y(a_w) = (Y_o + ka_w)/\{1 + \exp[(a_w - a_{wc})/b]\} \quad (3)$$

or

$$Y(a_w) = (Y_o - Y_r + ka_w)/\{1 + \exp[(a_w - a_{wc})/b]\} + Y_r \quad (4)$$

where again Y_o is the intercept of $Y(a_w)$; k is a constant (roughly the slope of the linear region); and Y_r is the residual level of $Y(a_w)$ where appropriate.

The effect of a_w on different properties can be classified by simply observing which pattern emerges (Fig. 1). It can also be quantified in terms of the model's parameters, notably a_{wc} and b (Wollny and Peleg 1994).

Thus, a significant discrepancy in the values of a_{wc} entails that the plasticization (with respect to properties in question) occurs at a different a_w or moisture content and, hence, temperature. Similarly, a significant discrepancy in the magnitude of b entails that there is a difference in the transition sharpness. The magnitude of

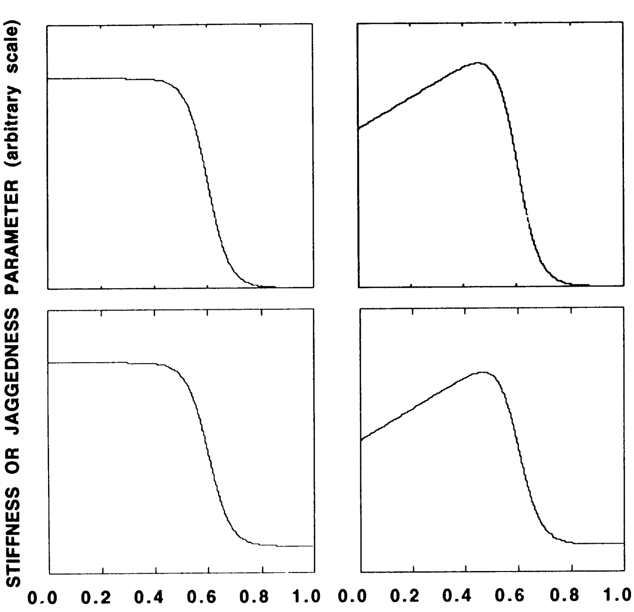

Fig. 1. Schematic view of different possible ways in which water activity can affect different mechanical properties of brittle cereal snacks. Top left: unmodified Fermi model (Eq. l); top right: shifted Fermi model (Eq. 2); bottom left: superimposed linear and Fermi models (Eq. 3;) and bottom right: shifted superimposed linear and Fermi models (Eq. 4).

k and Y_r can be an indicator of the deformation mechanism. But in the case of Y_r it can also be only an inevitable result of the parameter definition and therefore devoid of physical significance (Wollny and Peleg 1994).

MATERIALS AND METHODS

Samples Preparation

Two national brand cheese balls and commercial croutons of three different types were purchased at a local supermarket. They were selected for being representative of brittle cellular products made by different processes and because their shapes and dimensions do not vary to an extreme degree, which facilitated the data interpretation. Samples of these products were taken from the package and stored in evacuated desiccators over saturated solutions of LiCl, CH_3COOK, $MgCl_2$, K_2CO_3, $Mg(NO_3)_2$, NaBr, $NaNO_2$, NaCl, and KCl at an ambient temperature of 25°C. This corresponds to a_w levels of about 0.11, 0.23, 0.33, 0.43, 0.52, 0.57, 0.65, 0.75, and 0.85, respectively (Greenspan 1977). Five specimens from each desiccator were removed and tested after 48 hr, a time found sufficient to reach a constant weight and, hence, practical equilibrium in preliminary experiments.

Mechanical Testing

Each specimen was compressed with an Instron universal testing machine (UTM) (model 1000, Instron Corp., Canton, MA) interfaced with a Macintosh II microcomputer through a Strawberry Tree interface card. A program written by Mark D. Normand was used to operate the instrument and to collect and process the data as described by Barrett et al (1992) and Rohde et al (1993a,b). The crosshead speed in all the experiments was 10 mm min^{-1} and the data retrieval rate 10 points sec^{-1} that is 60 points per mm of deformation. The average diameter of cheese balls and cheese puffs was ≈17 and 14 mm, respectively. The side of the approximately cubic croutons was ≈12 mm. All the specimens were compressed to ≈60% of their original diameter.

Data Processing

The voltage-time data files were converted to force ($F(\varepsilon)$) vs. engineering strain (ε) relationships using the UTM's sensor conversion constant. (Because of the imperfect morphology and nonuniform structure, the stress cannot be meaningfully determined). The F vs. ε files were fitted with a fourth degree polynomial model:

$$F(\varepsilon) = k_o + k_1\varepsilon + k_2\varepsilon^2 + k_3\varepsilon^3 + k_4\varepsilon^4 \quad (5)$$

using the Systat program. The force values at 20 and 30% deformation ($\varepsilon = 0.2$ and 0.3, respectively) were recorded and considered as two empirical measures of stiffness for the purpose of verification (Wollny and Peleg 1994).

A normalized dimensionless mechanical signature of each specimen $Y(\varepsilon)$ was created by the transformation:

$$Y(\varepsilon) = [F(\varepsilon) - F^*(\varepsilon)]/F^*(\varepsilon) \quad (6)$$

where $F^*(\varepsilon)$ is the fitted value using Eq. (5).

These mechanical signatures (strain range of $0.1 < \varepsilon < 0.6$) were subjected to the blanket algorithm (Peleg et al 1984) to determine their apparent fractal dimension using the procedure described by Normand and Peleg (1988) and Rohde et al (1993b). The elimination of the data corresponding to $\varepsilon < \sim 0.1$ is necessary to avoid artifacts resulting from the use of Eq. 5, where very large values of $Y(\varepsilon)$ can be produced when $F^*(\varepsilon)$ has a very small magnitude. The truncation, however, has only a very minor effect on the calculated jaggedness parameters (Barrett et al 1992). The procedure is based on "coating" the normalized signature with successive layers of a "blanket" following certain mathematical rules and calculation of the corresponding length by dividing the area of the coated signature after each iteration by its thickness. The apparent fractal dimension (D_f) of the signature is calculated from the slope of the linear region of the corresponding Richardson plot, that is the plot of the length versus the blanket's half thickness in logarithmic coordinates. The apparent fractal dimension is a jaggedness measure on a scale from 1.0 (corresponding to a smooth [Eucledian] signature) to 2.0, the theoretical upper limit where the signature is so jagged as to almost completely fill the area on which it is drawn. (An area has a Eucledian dimension of two by definition). The program for calculating the fractal dimension using the procedure was written by Mark D. Normand.

To verify the jaggedness assessment through the fractal analysis by an independent test, the normalized signature was also transformed into a power spectrum of 15 harmonics (Ramirez 1985) using the fast Fourier transform which is standard option of the Systat package. The mean magnitude of the resulting power spectrum (Rohde et al 1993a, Wollny and Peleg 1994) was used as the added independent measure of jaggedness.

RESULTS AND DISCUSSION

Experimental and normalized force-deformation curves of two types of cellular brittle cereal foods and the corresponding power spectra are shown in Figures 2–7. The figures illustrate that the exposure to a moist atmosphere results not only in changes of stiffness, that is the overall force level, but also in the jaggedness of the force-deformation itself. The latter is particularly evident in the general appearance of the normalized curves or signatures

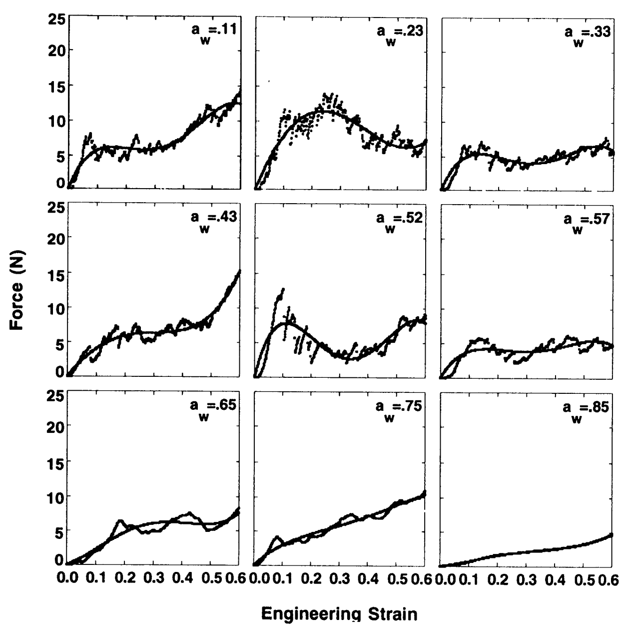

Fig. 2. Examples of force-deformation curves of cheese puffs at various water activity levels.

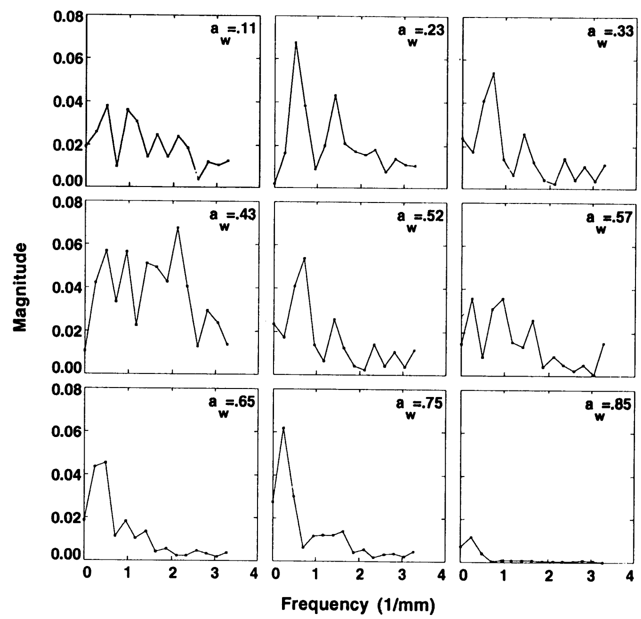

Fig. 4. Examples of the Fourier power spectrum of cheese puffs at various water activity levels.

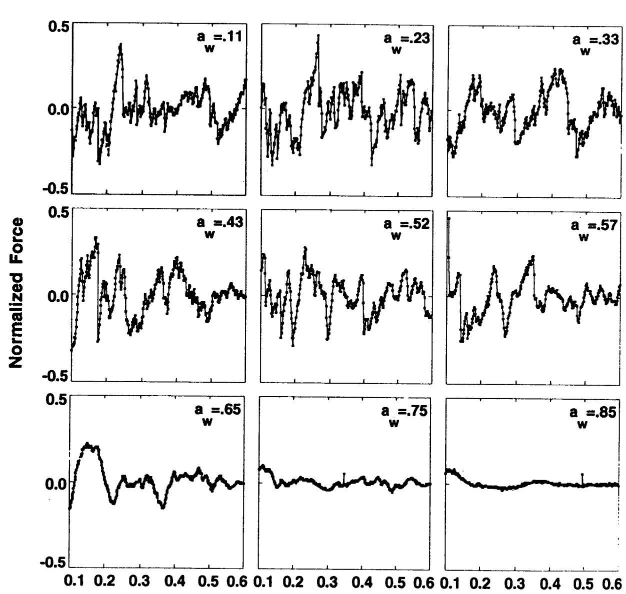

Fig. 3. Examples of normalized mechanical signatures of cheese puffs at various water activity levels.

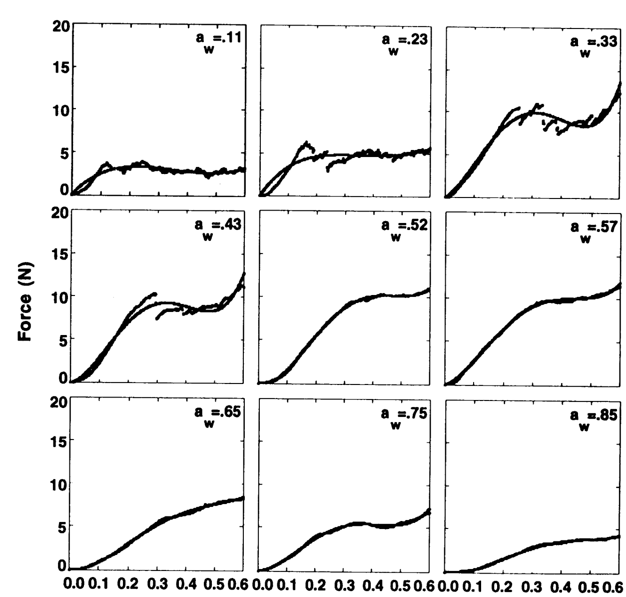

Fig. 5. Examples of force-deformation curves of French bread croutons at various water activity levels.

(Figs. 3 and 6) and their power spectra (Figs. 4 and 7). The power spectra clearly demonstrate that the effect is primarily due to the disappearance of the high frequencies fluctuations and to a lesser extent to the general shape of curves themselves, which is primarily manifested in the low frequency region. Similar effects were observed in all the materials tested in this work and is in agreement with previous reports (Barrett et al 1992, Rohde et al 1993, Wollny and Peleg 1994). As can be seen in Figures 2 and 5, the fourth degree polynomial model used to calculate the normalized signature had an adequate fit and captured the general shape of each force-deformation curve. The results would hardly be affected had an alternative polynomial or even another type of a mathematical model been used, as shown by Barrett et al (1992) and Nuebel and Peleg (1993).

Because of the irregular shape of the force-deformation curve and the imperfect geometry of the individual specimens, determination of a meaningful stress-strain relationship and calculation of a deformability modulus from it is not an easy task. Consequently, the stiffness of each particulate, which expresses its resistance to deformation, was monitored as the fitted force at two predetermined deformations 20 and 30%. Although there are other alternatives, the fitted rather than the actually recorded value appears to be a more representative index of stiffness. This is because the fitting procedure provides an element of averaging around the selected deformation level. The selection of two deformation levels, as already mentioned, provides a sort of internal control, and if the fitted values in the two show the same trend, one can more safely treat it as a true manifestation of actual changes in stiffness unaffected by computational artifacts. A similar argument holds in considering the jaggedness of the normalized signature as a measure related to crunchiness (Wollny and Peleg 1994). Thus, the jaggedness is also expressed by two parameters: the apparent fractal dimension D_f and the mean magnitude of the power spectrum M_m. Since the effect of a_w on the power spectrum was quite dramatic, filtering of the low frequencies was unnecessary.

Plots of stiffness and jaggedness parameters versus a_w relationships are demonstrated in Figures 8–13. These regression parameters are listed in Tables I and II. The most salient feature of the relationships is that, despite the considerable scatter, there was a generally good agreement between the two stiffness parameters (Figs. 8, 10, and 12) and between the two jaggedness parameters (Figs. 9, 11, and 13) but not necessarily between the two types (Figs. 8 vs. 9, 10 vs. 11, and 12 vs. 13). The latter was manifested in a different overall shape and, hence, in the mathematical model as well as in the magnitude of the characteristic parameters (Tables I and II). This is a strong indication that the two mechanical properties, stiffness and brittleness or crunchiness, need not change in unison as a result of moisture sorption. It is in agreement with similar such observations already mentioned (Attenburrow and Davis 1993, Wollny and Peleg 1994) and adds support to the notion that glass transition in a complex food system is a continuous process with different manifestations rather than a dramatic event in which all properties change abruptly and

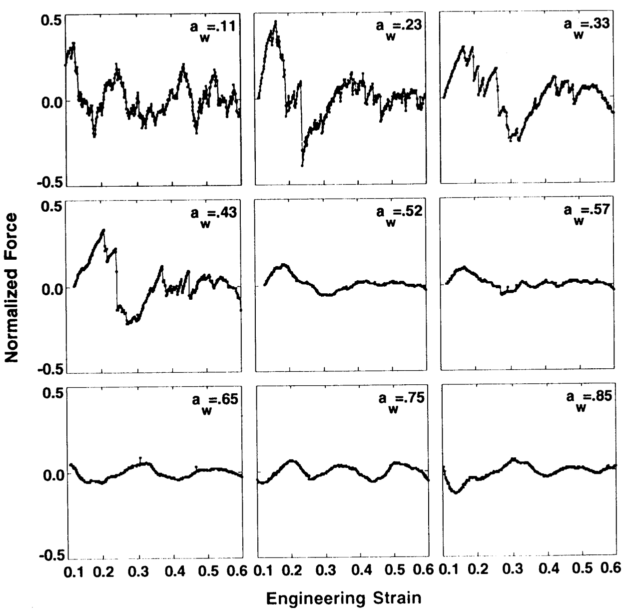

Fig. 6. Examples of normalized mechanical signatures of French bread croutons at various water activity levels.

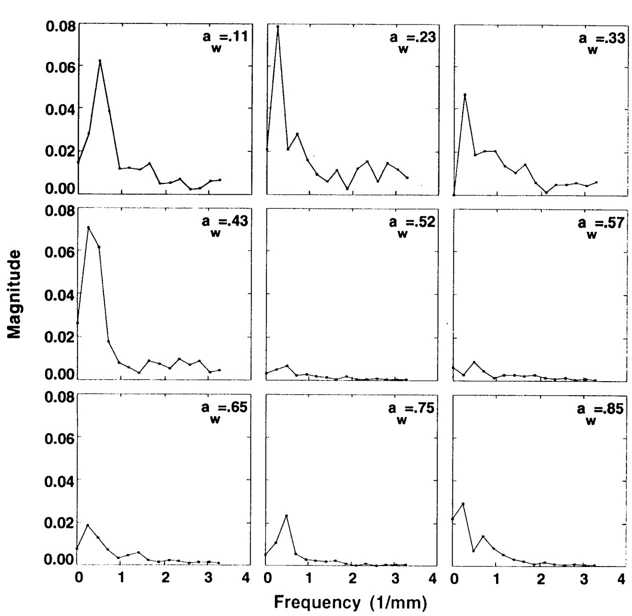

Fig. 7. Examples of the Fourier power spectrum of French bread croutons at various water activity levels.

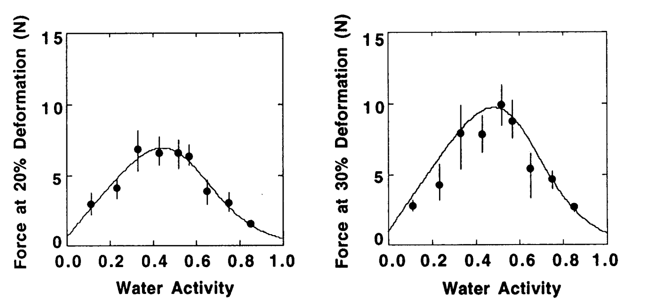

Fig. 8. Effect of water activity on the stiffness parameters of cheese puffs. Vertical bars mark the parameters magnitude range.

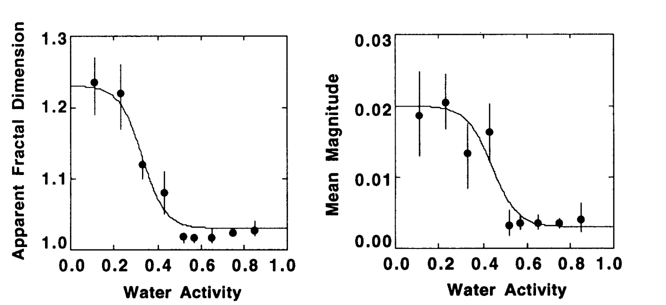

Fig. 9. Effect of water activity on the jaggedness parameters of the normalized signatures of cheese puffs. Vertical bars mark the parameters magnitude range.

simultaneously.

The jaggedness parameters versus a_w relationships could be adequately described by the standard or shifted Fermi equation as shown in the figures and Table II. This enabled quantitative characterization of the a_w effects in terms of the constants a_{wc} and b. It can clearly be seen from Table II, that in the two puffed extrudates that contained cheese, crunchiness was lost at $a_w \approx 0.3–0.45$; in the croutons, crunchiness was lost at ≈0.5–0.7. There were also minor differences in the steepness parameter b but their significance is not clear.

The stiffness parameters versus a_w relationships were of different types. The most notable (Figs. 8 and 10) was characterized by an apparent increase in stiffness as the a_w rose from 0.11 to ≈0.5 in one case, and to ≈0.6 in the other. In either case, there was a substantial drop at higher a_w levels, irrespective of whether there was a peak or not (Fig. 12). Data showing an apparent peak stiffness was also reported by others (Attenburrow and Davis 1993). The agreement between the results with the two stiffness measures F (20%) and F (30%) in all the cases excludes the possibility that it was an accidental observation or an artifact. Because water acts as a plasticizer and is therefore expected to lower a material's stiffness, the observation of a "stiffness peak" requires an explanation. The force at any given deformation is a measure of the specimen's mechanical integrity at that particular deformation. At the very low a_w levels, the structure of cellular cereal products is extremely brittle and hence very fragile. This structure collapses very rapidly, and its destroyed elements offer no resistance to added deformation. Moreover, failure propagates very quickly in such a structure and causes its fragmentation and disintegration. It is highly plausible that at moderate levels of a_w, the partially plasticized matrix is more cohesive and does not disintegrate as readily. Consequently, more structural elements remain intact, thus offering more resistance which is expressed in a higher apparent stiffness. Consistent with this explanation is the observation that there was never a case where the jaggedness parameters, which are measures of brittleness, had anything even remotely reminiscent of a peak, irrespective of the material type. It is also supported by the observation of Nicholls et al (1995), who reported that the strain at failure can reach a maximum at an intermediate degree of plasticization. Since the area under the force-deformation curve is associated with toughness (i.e., the absorbed energy), one can also conclude that in those materials where a peak force is recorded, partial plasticization is accompanied by toughening.

As can be seen in Tables I and II, the magnitude of the Fermi or modified Fermi's models constants varied considerably among the tested materials. This is a reflection of structural and compositional differences that were not determined in this work. It can be argued though that global mechanical parameters such as stiffness and jaggedness are determined simultaneously by the cellular characteristics of the matrix (e.g., open or closed cells, cell size distribution, cell wall thickness) and the mechanical properties of the cell wall material and its moisture dependency. Or, in other words, the actual mechanical behavior of cellular cereal products of the kind tested are determined by phenomena at two different levels (at least): the molecular and the structural and their interactions. Because the latter are regulated by the specific chemistry of the components and the specific structural characteristics, it was not surprising that the products' mechanical behavior, when dry and after moisture sorption, differed. Thus, one can conclude that, although certain general patterns of mechanical behavior can be expected, the detailed pattern cannot be predicted on the basis of either the composition or the structure alone.

CONCLUSIONS

Moisture sorption affects different mechanical properties of brittle cellular cereal foods in different ways. Those that are related to brittleness or crunchiness can be described in terms of the original or shifted Fermi equation, whose parameters can be used to indicate at what a_w level these attributes are lost and what is the a_w span at which the loss occurs. Changes of stiffness as a result of moisture sorption can (but need not always) assume a

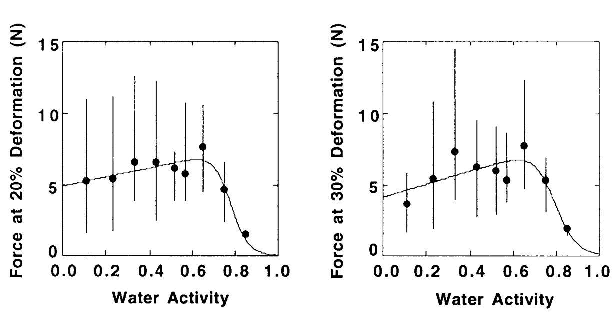

Fig. 10. Effect of water activity on the stiffness parameters of French bread croutons. Vertical bars mark the parameters magnitude range.

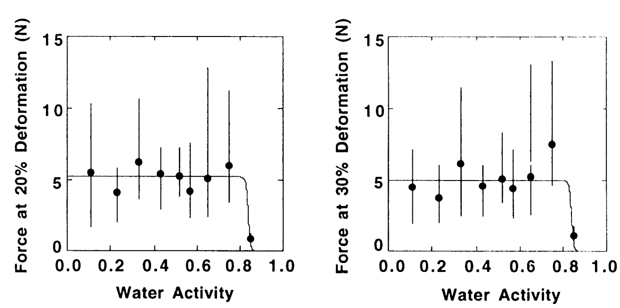

Fig. 12. Effect of water activity on the stiffness parameters of pumpernickel croutons. The vertical bars mark the parameters magnitude range.

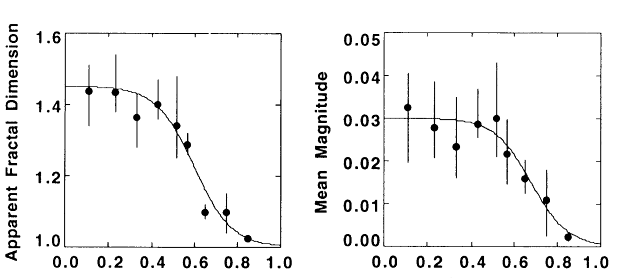

Fig. 11. Effect of water activity on the jaggedness parameters of the normalized signatures of French bread croutons. The vertical bars mark the parameters magnitude range.

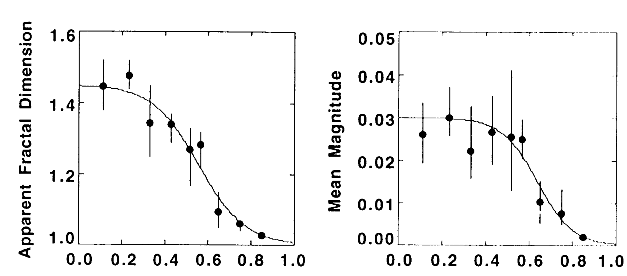

Fig. 13. Effect of water activity on the jaggedness parameters of the normalized signatures of pumpernickel croutons. The vertical bars mark the parameters magnitude range.

TABLE I
Effect of Water Activity (a_w) on Two Stiffness Parameters of Selected Brittle Cereal Foods[a]

Material	Force at 20% Deformation F_o (N)	k (N)	a_{wc} (–)	b (–)	r^2 (–)	Force at 30% Deformation F_o (N)	k (N)	a_{wc} (–)	b (–)	r^2 (–)
Cheese balls	0.35	11	0.62	0.087	0.998	0	14	0.64	0.090	0.985
Cheese puffs	0.65	19	0.58	0.110	0.985	1.0	23	0.63	0.110	0.992
Crutons										
French bread	4.9	3.2	0.78	0.040	0.990	4.1	4.6	0.79	0.050	0.976
Pumpernickel	5.2	0	0.84	0.005	0.980	5.0	0	0.84	0.005	0.955

[a] Based on the modified Fermi equation: $F(a_w) = F_o + ka_w)/\{1 = \exp[(a_w - a_{wc})/b]\}$ where $F(a_w)$ is the magnitude of the mechanical parameter; F_o is its magnitude in the dry state; k is a constant (roughly the slope of the linear region); a_{wc} roughly marks the inflection point of $F(a_w)$ b is a constant that accounts for the steepness of the relationships at a_{wc}.

TABLE II
Effect of Water Activity (a_w) on Two Jaggedness Parameters of the Normalized Mechanical Signatures of Selected Brittle Cereal Foods[a]

Material	Apparent Fractal Dimension (D_f) D_{fo}	D_{fr}	a_{wc}	b	r^2	Mean Magnitude of the Power Spectrum (M_m) M_{mo}	M_{mr}	a_{wc}	b	r^2
Cheese balls	1.40	1.04	0.33	0.058	0.999	0.027	0.006	0.33	0.071	0.998
Cheese puffs	1.23	1.03	0.33	0.052	0.999	0.020	0.003	0.44	0.057	0.959
Croutons										
French bread	1.45	1.00	0.60	0.088	0.999	0.030	0	0.68	0.085	0.984
Pumpernickel	1.45	1.00	0.56	0.110	0.999	0.030	0	0.64	0.082	0.974

[a] Based on the modified Fermi equations: $D_f(a_w) = (D_{fo} - D_{fr})/\{1 + \exp[(a_w - a_{wc})/b]\} + D_{fr}$ and $M_m(a_w) = (M_{mo} - M_{mr})/\{1 + \exp[(a_w - a_{wc})/b]\} + M_{mr}$, respectively, where D_{fo} is the apparent fractal dimension at $a_w = 0$; D_{fr} is its "residual" or asymptotic magnitude; M_{mo} is the mean magnitude of the power spectrum at $a_w = 0$; M_{mr} is its "residual" or asymptotic magnitude. a_{wc} marks the inflection point of $D_f(a_w)$ or $M_m(a_w)$, the transition center, and b is a constant that accounts for the steepness of the relationships at a_{wc}.

more complicated pattern with an observed apparent stiffness peak at a moderate a_w levels. This peak is definitely not an experimental artifact. It is most probably a result of partial plasticization of the cell wall material, which increases the structure's cohesion and, hence, toughness. Thus, structural elements that would have been completely destroyed and eliminated as a result of brittle failure at very low a_w levels can remain intact or only partly destroyed and continue to offer resistance.

Description of the relationship between stiffness and a_w of toughening cereal products requires modification of the Fermi model. An added linear term to the equation numerator is sufficient to account for the stiffness increase before continued plasticization weakens the structure and reduces the forces that it can resist.

ACKNOWLEDGMENT

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